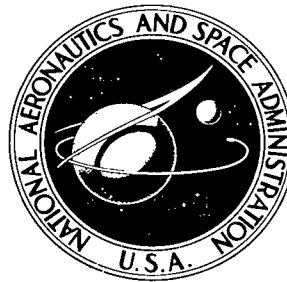


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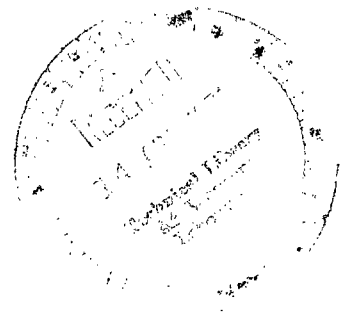


BOUNDARY LUBRICATION AND THERMAL STABILITY STUDIES WITH FIVE LIQUID LUBRICANTS IN NITROGEN TO 400° C

by William R. Jones, Jr., and William F. Hady

Lewis Research Center

Cleveland, Ohio 44135



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16. Abstract <p>A pin-on-disk friction apparatus was used to determine friction and wear of steel in nitrogen with five lubricants. Good boundary lubrication was obtained from 200° to 400° C (392° to 752° F) with a glycol derivative and with a synthetic paraffinic oil containing an antiwear additive. Equally good results were obtained from 200° to 320° C (392° to 608° F) with an advanced ester. A paraffinic resin and a synthetic paraffinic oil without an additive were effective boundary lubricants at some temperatures but not over the entire temperature range. Maximum useful lives calculated from thermal stability data were usually in the order of paraffinic resin > synthetic paraffinic oil > glycol >> advanced ester at temperatures from 200° to 350° C (392° to 662° F).</p>					
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BOUNDARY LUBRICATION AND THERMAL STABILITY STUDIES WITH FIVE LIQUID LUBRICANTS IN NITROGEN TO 400° C

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SUMMARY

A pin-on-disk sliding friction apparatus was used to determine friction and wear of steel in dry nitrogen (<20 ppm H_2O) with five liquid lubricants. The lubricants were (1) a superrefined light paraffinic resin, (2) a synthetic paraffinic oil with an antiwear additive, (3) a synthetic paraffinic oil without an antiwear additive, (4) an advanced ester, and (5) a glycol derivative. Experimental conditions were a 1-kilogram load, a 100-rpm disk speed, a 17 meter per minute surface speed, a 200° to 400° C (392° to 752° F) disk temperature range, and a 1-hour test duration. Test specimens were made of consumable electrode vacuum melted (CVM) M-50 tool steel. In addition, useful lives of each lubricant were calculated at several temperatures from thermal stability data using an Arrhenius rate law.

Good boundary lubrication was obtained over the entire temperature range with a glycol derivative and a synthetic paraffinic oil containing an antiwear additive. Equally good results were obtained from 200° to 320° C (392° to 608° F) with a formulated advanced ester. The other fluids (paraffinic resin and synthetic paraffinic oil without an additive) were effective boundary lubricants at some temperatures but not over the entire temperature range.

Thermal stability calculations (based on 10 percent decomposition) were used to determine maximum useful lives for the four lubricants in nitrogen at 200°, 250°, 300°, and 350° C (392°, 482°, 572°, and 662° F). Over most of the temperature range, the order of calculated useful lives was paraffinic resin $>$ synthetic paraffin $>$ glycol derivative $>>$ advanced ester.

INTRODUCTION

Higher temperature requirements for lubricants are a direct consequence of higher

output engines and increases in flight speeds. Bulk oil temperatures to 260°C (500°F) and bearing temperatures to 400°C (752°F) are predicted for advanced aircraft (refs. 1 to 3). Lubricants for use at these temperatures in air must possess good oxidation and thermal stability. Because sliding and skidding can occur in rolling element bearings, these lubricants must also possess good boundary lubricating ability.

Lubricant oxidation usually occurs before thermal breakdown and is the limiting factor. Nitrogen inerting of aircraft lubricating systems (refs. 2 and 4) prevents oxidation and can extend the maximum temperature limit of many lubricants by more than 100°C (212°F). Maximum temperature limits from manufacturers' data in air and inerted systems for three typical lubricants appear in figure 1.

In an inerted system where oxidation is no longer possible, thermal stability becomes the limiting factor. One of the common methods of determining the thermal stability of lubricants is by measuring the isothermal time rate of increase of vapor pressure at various temperatures. The instrument used for these determinations is an isoteniscope based on an original design of Smith and Menzies (ref. 5). Blake and Hamman used a modified isoteniscope for thermal stability studies (ref. 6). Recently, Fowler and Trump (ref. 7) have developed an automated tensimeter which eliminates the time consuming manual procedures of earlier isotenisscopes. The isoteniscope determines the thermal decomposition temperature and the activation energy for decomposition for organic fluids. From these two properties, one may calculate maximum useful lives for lubricants assuming a certain tolerable percent decomposition.

The objectives of this investigation were (1) to determine the friction and wear of CVM M-50 steel in dry nitrogen with five liquid lubricants using a pin-on-disk sliding friction apparatus from 200° to 400°C (392° to 752°F), (2) to determine the thermal decomposition temperatures and activation energies for decomposition of the lubricants using a tensimeter, and (3) to calculate the maximum useful lives of the lubricants from the thermal stability data.

These lubricants (table I) were (1) a superrefined paraffinic resin, (2) a synthetic paraffinic oil with an antiwear additive, (3) a synthetic paraffinic oil without an antiwear additive, (4) an advanced ester, and (5) a glycol derivative. Experimental conditions included a 1-kilogram load, a 100 rpm disk speed, a 17 meter per minute surface speed, a 200° to 400°C (392° to 752°F) disk temperature range, a 1-hour test duration, and a dry nitrogen atmosphere.

SYMBOLS

A' frequency factor, torr/sec

E' activation energy, kcal/mole

M	molecular weight
$(dP/dt)_T$	isothermal rate of vapor pressure rise at temperature T, torr/sec
p	pressure, torr
R	gas constant, cal/mole-K
T	absolute temperature, K
T_D	thermal decomposition temperature, °C (°F)
t	time, hr
x	percent decomposition

APPARATUS

Pin on Disk

The friction and wear apparatus used in this investigation is shown in figure 2. The test specimens, hemispherically tipped riders having 0.476-centimeter radii and 6.3-centimeter diameter disks, are mounted inside a stainless steel chamber (3.7-liter volume) where the atmosphere is controlled with respect to oxygen and moisture content.

The disk specimen is partially submerged in a pyrex cup containing the test fluid with the rider specimen mounted on a gimbaled arm above the cup. Induction heating of the disk specimen permits transfer of heat to the lubricant which is partially cooled by means of the water passing through the heating coil. Test temperatures of the disk are monitored with an infrared pyrometer and the bulk lubricant temperature is measured with a thermocouple. In these studies, test temperatures from 200° to 400° C (392° to 752° F) permitted the bulk lubricant temperature to stabilize 100° to 150° C (212° to 270° F) below the disk temperature.

Dry nitrogen was obtained directly from compressed gas cylinders. The moisture content was monitored with a moisture analyzer with an accuracy of ±10 ppm. Oxygen content was monitored with an oxygen analyzer having an accuracy of ±10 ppm. Frictional force was measured with a strain gage and was recorded on a strip chart recorder.

Tensimeter

The tensimeter apparatus shown in figure 3 consists of two units: the sample oven with its associated temperature programmer and the electronics module containing the

recorder. The basic function of the tensimeter consists of heating a liquid sample and of plotting either the vapor pressure, or the isothermal rate of increase of vapor pressure (due to thermal decomposition), as a function of temperature.

The sample assembly appears in figure 4. The sample cell is a glass bulb having a 5-milliliter ($5 \times 10^{-6} \text{ m}^3$) volume, with a stem extending through the oven wall to a valve and pressure transducer mounted outside the oven.

PROCEDURE

Pin-on-Disk Apparatus

Disks and riders were made of consumable electrode vacuum melted (CVM) M-50 steel, disk hardness was Rockwell C 62-64, and rider hardness was 56 to 58. They were ground and lapped to a surface finish of 4 to 8 microinches rms (10×10^{-8} to $20 \times 10^{-8} \text{ m}$). Specimens were scrubbed with a paste of levigated alumina and water, rinsed first with tap water and then with distilled water, and placed in a desiccator.

Lubricants were degassed at 75°C (167°F) under a vacuum. Dissolved oxygen concentration was measured with a polarographic probe and found to be less than 5 ppm. No attempt was made to remove dissolved water.

The specimens were mounted in the test chamber and 70 milliliters ($7 \times 10^{-5} \text{ m}^3$) of lubricant were placed in the lubricant cup. The test chamber was purged with nitrogen for 10 minutes at a flow rate in excess of 50 liters per hour ($5 \times 10^{-2} \text{ m}^3/\text{hr}$). The disk was rotated at test speed while being heated to test temperature. After test temperature is reached, the rider is loaded against the disk with a 1-kilogram load (initial Hertz stress, $1 \times 10^9 \text{ N/m}^2$). The nitrogen flow rate was reduced to 35 liters per hour ($3.5 \times 10^{-2} \text{ m}^3/\text{hr}$), and 1 psig ($6.9 \times 10^3 \text{ N/m}^2$) pressure was maintained in the chamber.

Frictional force and bulk lubricant temperature were continuously recorded. Disk temperature was continuously monitored and manually controlled to $\pm 5^\circ \text{C}$ (9°F). Disk temperature calibration procedure is described in reference 8. Tests were 1 hour in duration, and rider wear scar diameter was recorded.

Tensimeter

Three to four milliliters (3×10^{-6} to $4 \times 10^{-6} \text{ m}^3$) of test fluid are placed in the sample cell. The cell is attached to the cell assembly and the fluid is degassed and refluxed under vacuum. The cell assembly is then placed in the oven. The sample is heated to an initial temperature about 50°C (122°F) below the suspected decomposition temperature. After a 5-minute stabilization period, the increase in vapor pressure, if any, is

recorded as a vertical bar during a fixed time interval. Then the programmer raises the sample temperature by a preset amount, usually 5°C (9°F), and the previous process is repeated. A typical plot appears in figure 5.

This is essentially a plot of the logarithm of the isothermal rate of vapor pressure increase as a function of reciprocal absolute temperature. A straight line is drawn connecting the tops of the recorded bars at the higher temperatures. The intersection with the temperature reference axis is the thermal decomposition temperature T_D . This temperature axis corresponds to a pressure rise of 50 torr per hour, which is the definition of the T_D . The activation energy for decomposition E' may be calculated from the slope of the straight line.

RESULTS AND DISCUSSION

Coefficient of Friction

The types of friction behavior observed during these experiments are illustrated in figure 6. In general, the synthetic paraffin with additive, the glycol derivative, and the advanced ester exhibited a low (<0.20) steady coefficient of friction (curve A). The synthetic paraffin without additive and the light resin exhibited a low coefficient of friction (<0.20) but with occasional erratic high friction (0.35 to 0.40) (curve B). Finally, the ester at 320°C (608°F) and the glycol at 400°C (752°F) exhibited low initial friction (<0.20) with an abrupt change to high friction (0.35 to 0.40) for the remainder of the test (curve C). This behavior clearly indicates lubrication failure due to loss of lubricant by evaporation. It was confirmed by the low fluid level after test completion. The coefficient of friction as a function of temperature for the unlubricated situation and for the five fluids appears in figures 7(a) to (e).

Wear-Temperature Behavior

Table II lists the arbitrary definitions used to describe wear rate levels. Rider wear as a function of temperature for unlubricated M-50 steel appears in figure 7(a). The results of the friction and wear tests are presented in figures 7(a) to (e) and in table III.

Paraffinic resin. - Rider wear as a function of temperature for the paraffinic resin appears in figure 7(b). Intermediate wear occurred from 200° to 400°C (392° to 752°F). Maximum wear occurs between 325° to 350°C (617° to 662°F).

Synthetic paraffin. - Rider wear as a function of disk temperature for the synthetic paraffin without and with additive appears in figure 7(c). For the fluid without additive, low wear occurs at both extremes of the 200° to 400°C (392° to 752°F) temperature

range; intermediate wear occurs from 220° to 375° C (428° to 707° F) and maximum wear occurs in the region 250° to 300° C (482° to 572° F). For the paraffin with additive, low wear was observed from 290° to 400° C (554° to 752° F). No difference in friction coefficient was observed for the two fluids.

Advanced ester. - Rider wear as a function of disk temperature for the advanced ester appears in figure 7(d). Low wear occurs from 200° to 320° C (392° to 608° F). Maximum wear occurred at 200° C (392° F). Lubricant evaporation at 320° C (608° F) prevented test completion.

Glycol derivative. - Rider wear as a function of temperature for the glycol derivative appears in figure 7(e). Low wear occurs from 200° to 400° C (392° to 752° F). A summary of the friction and wear test results for all fluids appears in table III.

Thermal Decomposition Temperature T_D

A common method of determining the thermal stability of organic compounds is by measuring time rate of vapor pressure rise with increasing temperature. The decomposition temperature is determined from a plot of the logarithm of the vapor pressure as a function of the reciprocal of absolute temperature. The temperature at which the vapor pressure curve deviates from linearity is taken as the decomposition temperature.

The decomposition temperature may also be determined from isothermal rates of pressure rise. Several rates are measured and the logarithm of rate of pressure rise is plotted as a function of the reciprocal of absolute temperature (fig. 5). The decomposition temperature is taken to be that temperature at which the rate of pressure rise is 50 torr per hour. This latter procedure was used to determine T_D in this report.

Thermal Stability Calculations

Useful lives for the four lubricants used in this study were calculated by using the equations developed by Blake and Hammann (ref. 6) at 200°, 250°, 300°, and 350° C (392°, 482°, 572°, and 662° F).

Reference 6 used an Arrhenius rate law for the thermal stability data correlation:

$$\frac{dP}{dt} = A'e^{-E'/RT} \quad (1)$$

The activation energy E' is calculated from

$$E' = -\frac{RT_1T_2}{T_1 - T_2} \ln \left[\left(\frac{dP}{dt} \right)_{T_2} \left(\frac{dP}{dt} \right)_{T_1} \right] \quad (2)$$

The two isothermal rates of pressure rise and the T_D are determined experimentally with a tensimeter. The thermal decomposition temperatures and activation energies for each lubricant appear in table IV. Two decomposition temperatures were determined for the ester. One was measured by the regular method and the second in the presence of iron powder. Thermal decomposition of esters is known to be catalyzed in the presence of steel (ref. 6). The decomposition of hydrocarbon and most other organic fluids appears to be unaffected by the presence of steel (ref. 6).

The useful life of each lubricant was calculated from

$$t = \frac{0.0285 T_D}{M} \log \left(\frac{100 - x}{100} \right) \text{antilog} \left[5 - 219 E' \left(\frac{1}{T_D} - \frac{1}{T} \right) \right] \quad (3)$$

Equations (1) to (3) were derived using the following assumptions:

- (1) Change of density with temperature was neglected.
- (2) Mode of decomposition of compound is unchanged from T_D to T .
- (3) Thermal decomposition is unimolecular.
- (4) One molecule enters the gas phase per each molecule decomposed.

Useful Lives

The useful life is defined as the time in hours required for 10-percent fluid thermal decomposition. The 10-percent value is purely arbitrary. Useful lives for the four lubricants as functions of temperature appear in figure 8. From 200° to 350° C (392° to 662° F) the order of decreasing useful lives was usually paraffinic resin > synthetic paraffin > glycol derivative >> advanced ester.

It is often assumed that a lubricant cannot or should not be used at temperatures above its T_D . Lubricants may be used above their T_D if one can tolerate their decreased useful lives (see fig. 8).

Another interesting feature shown in figure 8 is that the useful life at temperatures above the T_D is not necessarily in the same order as the T_D . At 350° C (662° F) the useful life for the ester ($T_D = 223^\circ \text{C}$ (433° F)) is 1.6 hours compared to 1.1 hours for the glycol ($T_D = 279^\circ \text{C}$ (534° F)). This could be quite important in a once through system where fluids may be subjected to temperatures well above their T_D .

Rider Wear as Function of T_D

In the boundary lubrication regime, the lubricating film is made up of many components. These can include physically and chemically adsorbed molecules, polymer films from the lubricant, and metal-lubricant organic and inorganic reaction films. It seems likely that a decrease in wear would be observed above the T_D because of the enhancement of some surface chemical reactions due to the formation of chemically reactive lubricant decomposition products. This appears to be true for most of the lubricants in this study. A decrease in rider wear occurs in the region of the T_D for the light resin and the synthetic paraffin without additive. A reduction in wear also is observed with the advanced ester near its catalyzed (in the presence of iron powder) T_D as well as its normal T_D . There appears to be no correlation between T_D and wear for the glycol derivative.

SUMMARY OF RESULTS

A pin-on-disk sliding friction apparatus was used to determine friction and wear of steel in dry nitrogen with five liquid lubricants. Disk temperature range was 200° to 400° C (392° to 752° F). Other conditions were a 1-kilogram load, a 17 meter per minute surface speed, and a 1-hour test duration. Test specimens were made of CVM M-50 steel. In addition, useful lives of each lubricant were calculated from thermal stability data. The major results were the following:

1. Low friction coefficient (<0.20) and low wear ($<10^{-12}$ m³/hr) occurred from 200° to 400° C (392° to 752° F) with the synthetic paraffinic oil with an antiwear additive and the glycol derivative. These conditions also existed from 200° to 320° C (392° to 608° F) with the advanced ester.
2. Low friction coefficient (<0.20) (with occasional bursts to 0.35 to 0.40) and intermediate wear ($>10^{-12}$ m³/hr) occurred over most of the temperature range with the paraffinic resin and the synthetic paraffinic oil without additive.
3. Maximum useful lives (based on 10-percent decomposition) calculated from thermal stability data were usually in the order of paraffinic resin $>$ synthetic paraffinic oil $>$ glycol derivative \gg advanced ester at temperatures from 200° to 350° C (392° to 662° F).

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, December 4, 1970,
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TABLE I. - PROPERTIES OF EXPERIMENTAL FLUIDS

Properties ^a	Lubricants				
	Superrefined paraffinic light resin	Synthetic paraffinic oil		Advanced ester	Glycol derivative
Additives	None	None	Antiwear	Oxidation	None
Kinematic viscosity, cS (m ² /sec)					
At 38° C (100° F)	750 (7.5×10 ⁻⁴)	440 (4.4×10 ⁻⁴)	440 (4.4×10 ⁻⁴)	30 (3.0×10 ⁻⁵)	120 (1.2×10 ⁻⁴)
At 99° C (210° F)	42 (4.2×10 ⁻⁵)	40 (4.0×10 ⁻⁵)	40 (4.0×10 ⁻⁵)	5.3 (5.3×10 ⁻⁶)	14 (1.4×10 ⁻⁵)
Pour point, °C (°F)	-6.7 (19)	-54 (-65)	-54 (-65)	-57 (-70)	-43 (-45)
Density at 20° C (68° F), g/cm ³	0.89	0.84	0.84	0.89	1.0
Thermal conductivity, J/(m)(sec)(K)	----	0.13	0.13	----	0.16
Specific heat at 38° C (100° F), J/(kg)(°C)	---	2000	2000	---	1900
Vapor pressure, ^b torr					
At 250° C (482° F)	35	<10	<10	30	---
At 300° C (572° F)	66	22	22	110	---

^aManufacturers' data.^bMeasured by authors.TABLE II. - ARBITRARY DEFINITIONS USED
TO DESCRIBE LEVELS OF WEAR RATE AND
COEFFICIENT OF FRICTION

Level	Coefficient of friction	Wear rate, m ³ /hr
Low	<0.20	<10 ⁻¹²
Intermediate	----	10 ⁻¹² to 10 ⁻¹¹
High	0.35 to 0.40	>10 ⁻¹¹

TABLE III. - SUMMARY OF FRICTION AND WEAR RESULTS

Disk temperature, °C	Unlubricated	Lubricant				
		Superrefined paraffinic light resin	Synthetic paraffinic oil with additive	Synthetic paraffinic oil without additive	Advanced ester	Glycol derivative
	Coefficient of friction					
200 (392)	0.63	0.10	0.12	0.12	0.14	0.12
250 (482)	.62	.11	.12	.12	.13	.10
300 (572)	.61	.12	.13	.13	.12	.08
350 (662)	.59	.12	.14	.14	----	.07
400 (752)	.57	.13	.14	.14	----	.06
Rider wear, m ³ /hr						
200 (392)	44.0×10 ⁻¹²	1×10 ⁻¹²	0.2×10 ⁻¹²	0.1×10 ⁻¹²	1×10 ⁻¹²	0.2×10 ⁻¹²
250 (482)	49.0	3	.9	5	0.3	.3
300 (572)	54.0	6	.6	7	.1	.4
350 (662)	59.0	6	.4	2	---	.5
400 (752)	64.0	1	.3	.6	---	.6

TABLE IV. - THERMAL DECOMPOSITION TEMPERATURES
AND ACTIVATION ENERGIES FOR FOUR LUBRICANTS

Lubricant	Thermal decomposition temperature, T _D , °C (°F)	Activation energy, E', kcal/mole
Synthetic paraffin	314 (597)	53
Paraffinic resin	312 (594)	51
Glycol derivative	279 (534)	48
Advanced ester	{ 258 (496) a 223 (433)	{ 33 a 23

^aIn the presence of Fe powder.

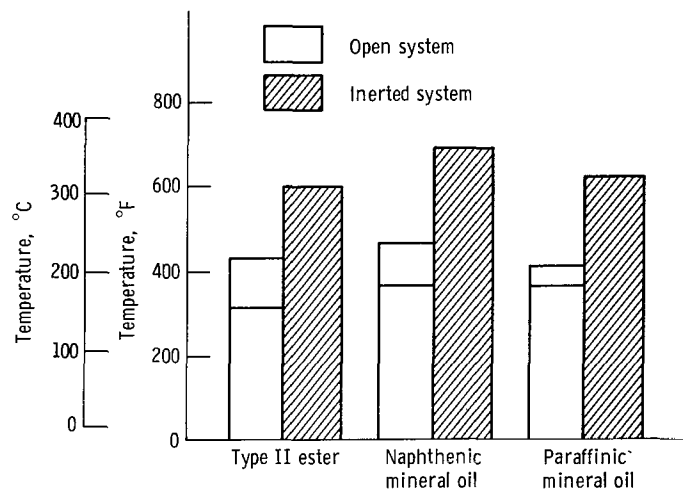


Figure 1. - Bulk lubricant maximum temperature limits from manufacturers' data. Open system limits based on oxidation-corrosion tests; inerted system limits based on isoteniscope tests.

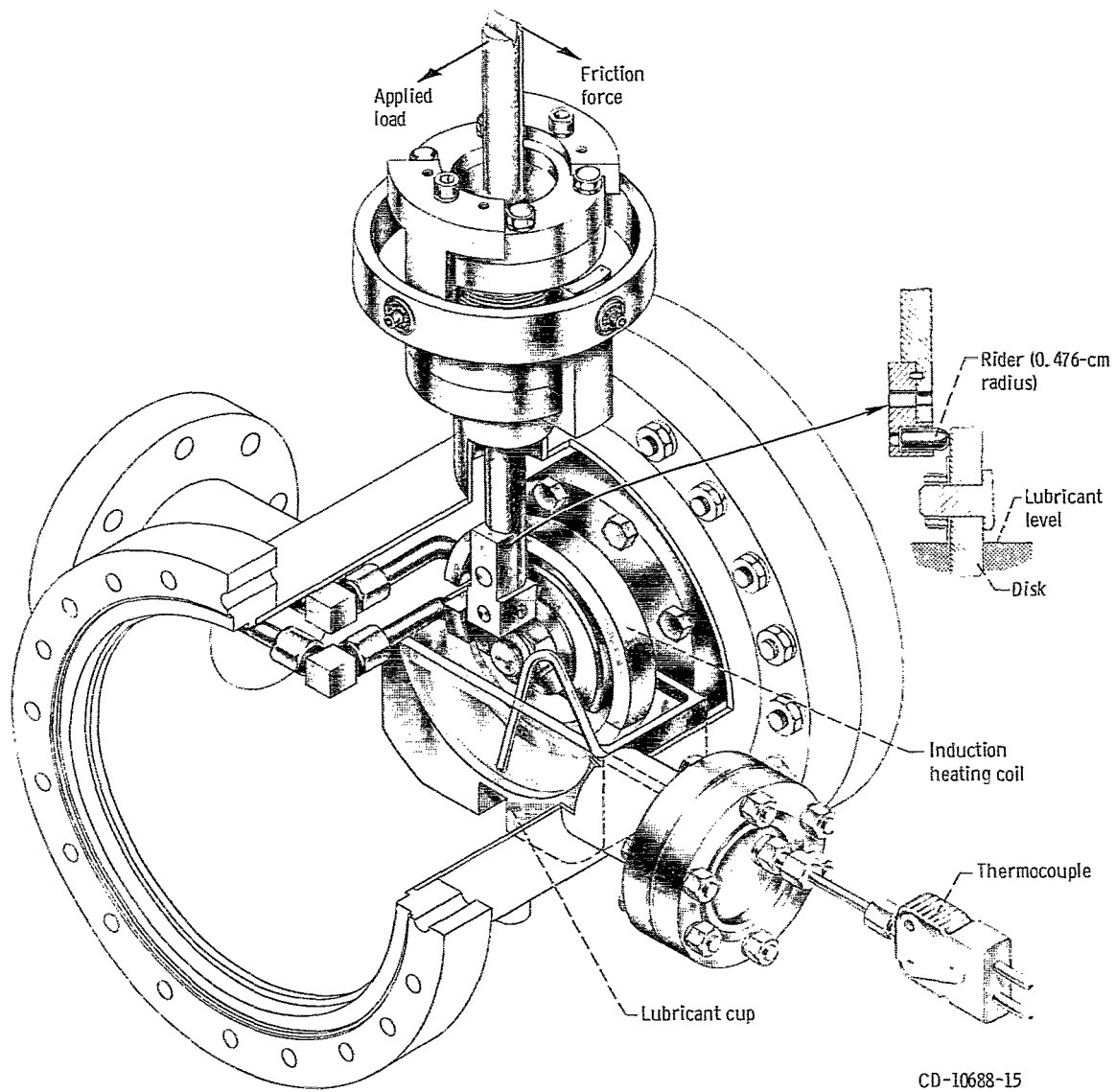


Figure 2. - Friction and wear apparatus.

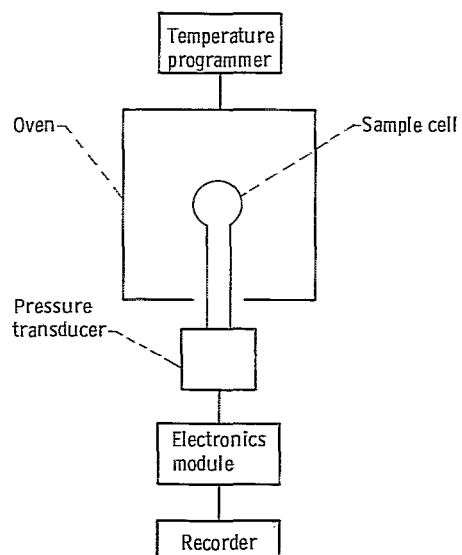


Figure 3. - Recording tensimeter.

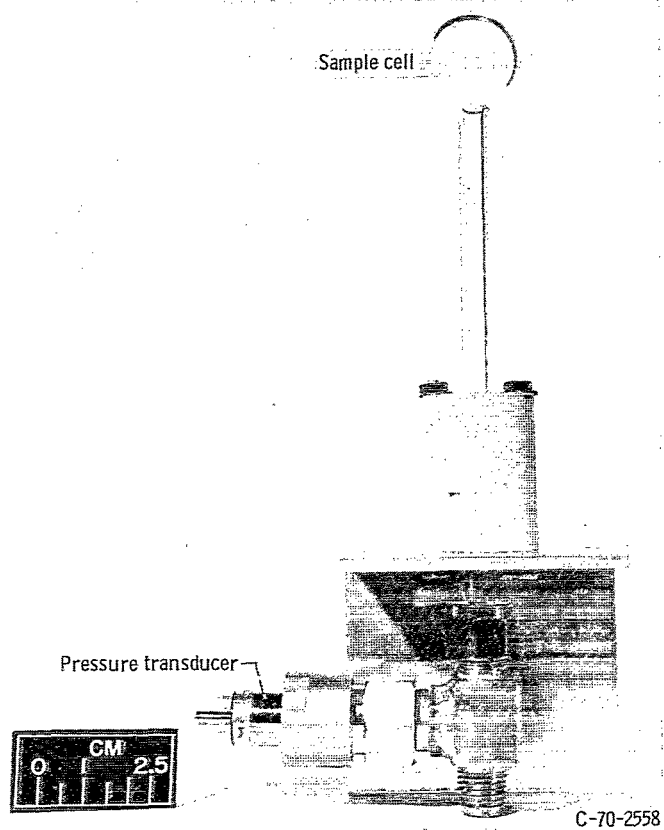


Figure 4. - Sample cell assembly.

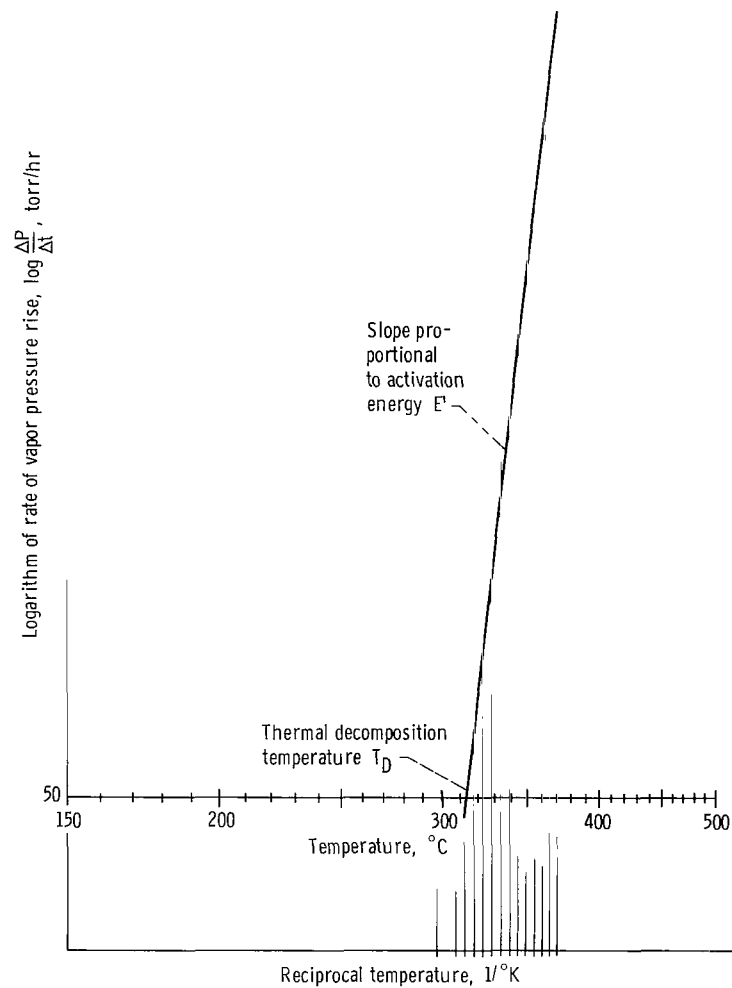


Figure 5. - Typical thermal decomposition curve logarithm of rate of vapor pressure rise as function of reciprocal of absolute temperature. Heating interval, 5° C (9° F).

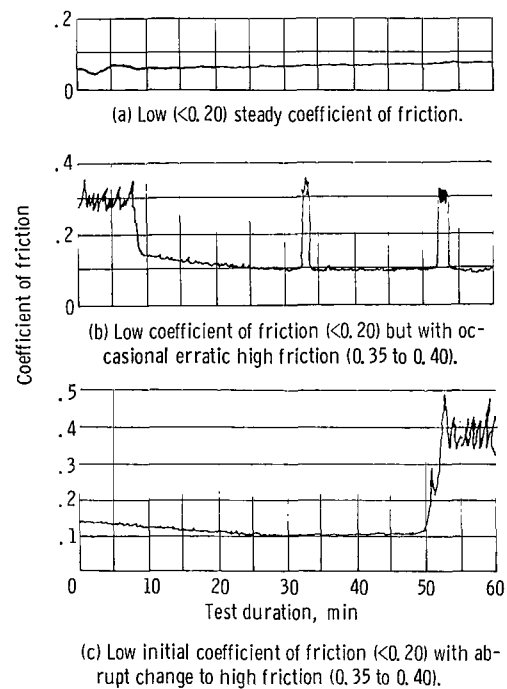
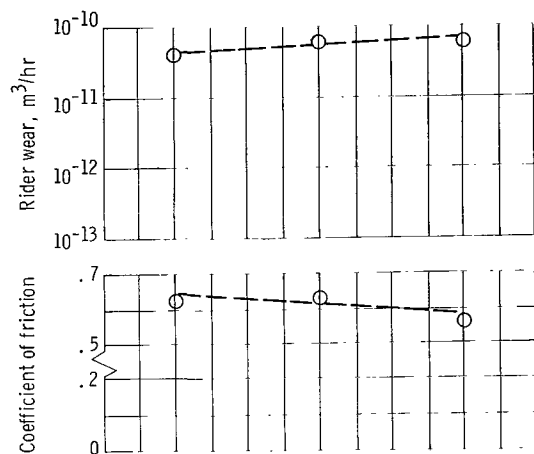
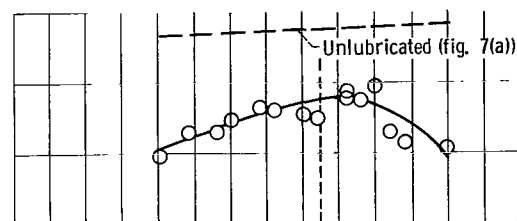


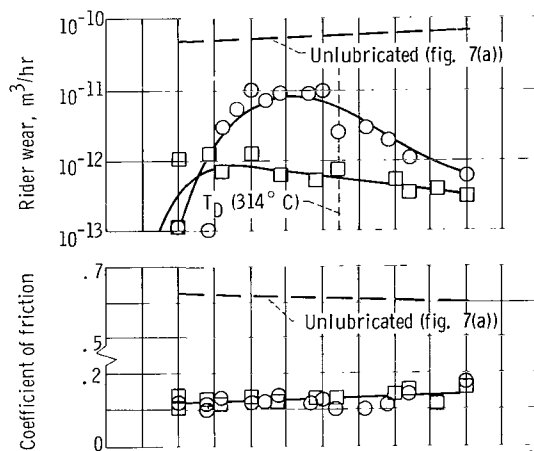
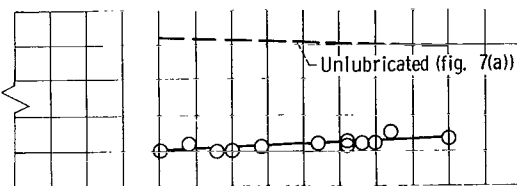
Figure 6. - Coefficient of friction as function of test duration.



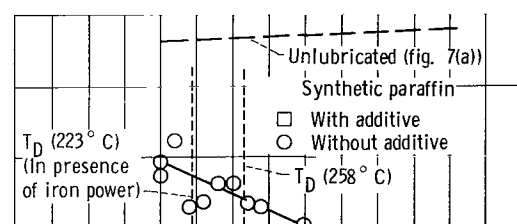
(a) Unlubricated M-50 tool steel.



(b) Superrefined light resin.



(c) Synthetic paraffin with and without antiwear additive.



(d) Advanced ester.

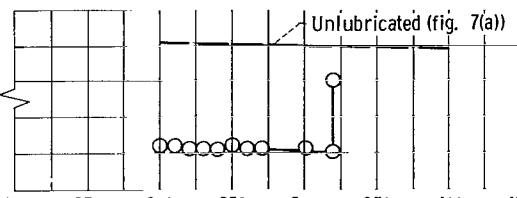
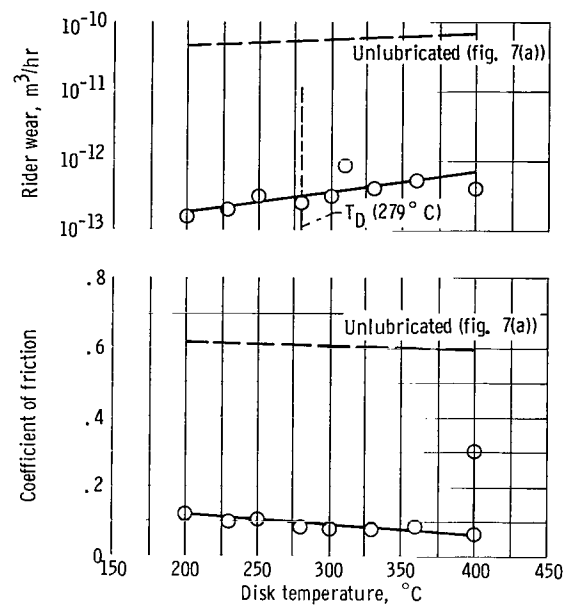


Figure 7. - Coefficient of friction and rider wear is function of disk temperature. Load, 1 kilogram; disk speed, 100 rpm; surface velocity, 17 meters per hour; dry nitrogen.



(e) Glycol derivative.

Figure 7. - Concluded.

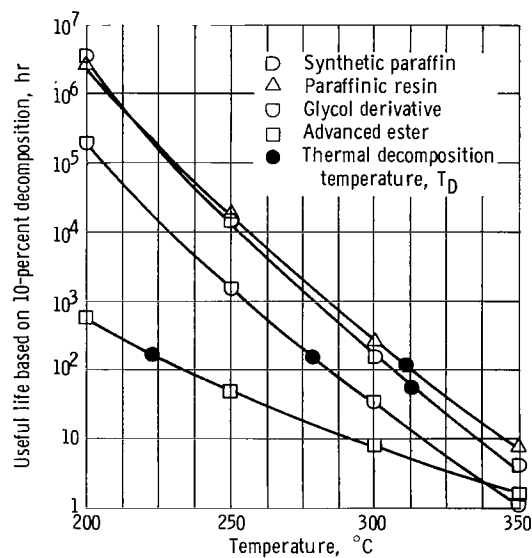


Figure 8. - Useful life (based on 10-percent decomposition) of four lubricants in nitrogen as function of temperature.

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